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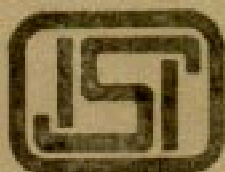
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*Indian Standard*

SPECIFICATION FOR  
PALLADIUM SALTS FOR ELECTROPLATING

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# *Indian Standard*

## SPECIFICATION FOR PALLADIUM SALTS FOR ELECTROPLATING

Electroplating Chemicals Sectional Committee, CDC 43

*Chairman*

DR H. V. K. UDUPA

*Representing*

Central Electrochemical Research Institute  
(CSIR), Karaikudi

*Members*

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Main Road, Malleswaram, Bangalore 560055 )

Central Electrochemical Research Institute  
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Bhabha Atomic Research Centre, Bombay

Hindustan Machine Tools ( Watch Division ),  
Bangalore

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Ronuk Industries Ltd, Bombay

# *Indian Standard*

## SPECIFICATION FOR PALLADIUM SALTS FOR ELECTROPLATING

### 0. FOREWORD

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 10 August 1981, after the draft finalized by the Electroplating Chemicals Sectional Committee had been approved by the Chemical Division Council.

**0.2** Electrodeposited palladium gives a greyish white coating. This coating, having hardness between those of gold and rhodium, is capable of taking fairly high, polish and remains tarnish-free even when subjected to a corrosive atmosphere or a temperature up to 350° C. Palladium is also used as an undercoat for rhodium in plating industry. The undercoat for palladium plating is usually silver or nickel.

**0.3** A number of palladium salts are used for electroplating this metal. Of these, palladium chloride (both anhydrous and dihydrate) and aminonitrite, aminonitrate and aminobromide of palladium are more common in use. Palladium chloride is used for strike plating of palladium before regular deposit of palladium. It is also used in electroless plating of the metal. Besides, palladium chloride and palladium aminobromide are used for heavy deposition of palladium [ more than 2.5 micrometers ( $\mu\text{m}$ ) ]. Palladium aminonitrite and aminonitrate are used for thin deposits ( between 2.5 to 5  $\mu\text{m}$  ) and are also used for preparing palladium sulphamate bath in conjunction with ammonium sulphate.

**0.4** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960\*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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### 1. SCOPE

**1.1** This standard prescribes requirements and methods of sampling and test for the following palladium salts for electroplating:

- a) Palladium chloride, anhydrous and dihydrate;

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\*Rules for rounding off numerical values (*revised*).

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- b) Palladium amminonitrite;
- c) Palladium amminonitrate; and
- d) Palladium amminobromide.

## 2. REQUIREMENTS

**2.1 Description** — Palladium salts shall conform to the following description:

| <i>Palladium salt</i>                       | <i>Description</i>                                                                                                                                                                                                                                                                                                                                                       |
|---------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Palladium chloride, anhydrous and dihydrate | Shall be in the form of dark red/garnet red/reddish brown needle like acicular crystals; highly hygroscopic, readily soluble in dilute hydrochloric acid ( with water it forms a basic insoluble salt ); free from foreign matter and visible impurities; and shall correspond essentially to the formula $\text{PdCl}_2$ or $\text{PdCl}_2 \cdot 2\text{H}_2\text{O}$ . |
| Palladium amminonitrite                     | Shall be in the form of almost colourless crystals, free from foreign matter and visible impurities; and shall correspond essentially to the formula $\text{Pd}(\text{NH}_3)_2(\text{NO}_2)_2$ .                                                                                                                                                                         |
| Palladium amminonitrate                     | Shall be in the form of almost colourless crystals; free from foreign matter and visible impurities; and shall correspond essentially to the formula $\text{Pd}(\text{NH}_3)_2(\text{NO}_3)_2$ .                                                                                                                                                                         |
| Palladium amminobromide                     | Shall be in the form of almost colourless, rhombic prism-like crystals; free from foreign matter and visible impurities; and shall correspond essentially to the formula $\text{Pd}(\text{NH}_3)_4\text{Br}_2$ .                                                                                                                                                         |

**2.2** Palladium salts for electroplating shall also comply with the requirements prescribed in Table 1 when tested according to the methods given in Appendix A. Reference to relevant clauses of Appendix A is given in col 7 of Table 1.

**TABLE 1 REQUIREMENTS FOR PALLADIUM SALTS FOR ELECTROPLATING**

( Clause 2.2 )

| Sl. No. | CHARACTERISTIC                                   | REQUIREMENTS       |                          |                          |                          | METHOD OF TEST<br>( REF TO CL No. OF APPENDIX A ) |
|---------|--------------------------------------------------|--------------------|--------------------------|--------------------------|--------------------------|---------------------------------------------------|
|         |                                                  | Palladium Chloride | Palladium Ammino-nitrite | Palladium Ammino-nitrate | Palladium Ammino-bromide |                                                   |
| (1)     | (2)                                              | (3)                | (4)                      | (5)                      | (6)                      | (7)                                               |
| i)      | Solubility                                       |                    | To pass the test         |                          |                          | A-2                                               |
| ii)     | Palladium ( as Pd ), percent by mass, <i>Min</i> | [ 59.0*<br>49.0†   | 40.5                     | 40.0                     | 30.0                     | A-3                                               |
| iii)    | Silver                                           |                    | To pass the test         |                          |                          | A-4                                               |
| iv)     | Lead                                             |                    | To pass the test         |                          |                          | A-5                                               |
| v)      | Copper                                           |                    | To pass the test         |                          |                          | A-6                                               |
| vi)     | Iron                                             |                    | To pass the test         |                          |                          | A-7                                               |

\* in anhydrous salt.

† in dihydrate salt.

**3. PACKING AND MARKING**

**3.1 Packing** — Palladium salts shall be packed in air-tight containers, preferably with a replaceable closure. The plater usually needs to withdraw a small quantity from the container without the danger of powder being scattered about. The container, therefore, should have an opening of such a size that powder may be withdrawn easily.

**3.2 Marking** — The containers shall be legibly and indelibly marked with the following:

- Name of the material,
- Net mass of the material and equivalent palladium content ( as Pd ) within brackets,
- Name of the manufacturer or his recognized trade-mark, if any; and
- Lot number and date of manufacture to enable the lot to be traced from the records.

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**3.2.1** The containers may also be marked with the ISI Certification Mark.

**NOTE**— The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

## **4. SAMPLING**

**4.1** The method of preparing samples of the material and the criteria for its conformity to this specification shall be as prescribed in Appendix B.

# **A P P E N D I X   A**

*( Clause 2.2 and Table 1 )*

## **ANALYSIS OF PALLADIUM SALTS FOR ELECTROPLATING**

### **A-1. Quality of Reagents**

**A-1.1** Unless specified otherwise, pure chemicals and distilled water ( *see* IS : 1070-1977\* ) shall be used in the tests.

**NOTE**— 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

### **A-2. TEST FOR SOLUBILITY**

**A-2.1 Procedure**— Weigh accurately about 1.0 g of the material and dissolve it in 50 ml of water. The material shall be taken as having satisfied the requirement of the test if there is no turbidity. Preserve the solution for further tests.

**NOTE**— For palladium chloride test solubility in dilute hydrochloric acid, as with water it forms a basic insoluble salt.

### **A-3. DETERMINATION OF PALLADIUM**

**A-3.0 Outline of the Method**— The sample is dissolved in water containing a few drops of nitric acid. Palladium is then precipitated as palladium dimethylglyoxime, filtered, dried and weighed.

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\*Specification for water for general laboratory use ( *second revision* ).

**A-3.1 Reagents****A-3.1.1 Dilute Nitric Acid** — Approximately 2 *N*.**A-3.1.2 Dilute Ammonium Hydroxide Solution** — 1 : 4 ( *v/v* ).**A-3.1.3 Dimethylglyoxime Solution** — 1 percent ( *m/v* ). Dissolve 1 g of dimethyl glyoxime in 100 ml of absolute alcohol.**A-3.1.4 Absolute Alcohol**

**A-3.2** To the solution reserved in **A-2.1** add a few drops of dilute nitric acid. Boil the solution till all nitrous fumes are expelled. Cool and dilute to about 100 ml with water. Add dilute ammonium hydroxide to make the solution alkaline, heat on a water bath for a few minutes and add dimethylglyoxime solution with stirring. Allow to stand on a water bath and add a drop of dimethylglyoxime solution again to ensure that precipitation is complete. Digest on a water bath for 1 h and then filter through a sintered glass crucible ( G No. 4 ). Wash the precipitate with water containing a few drop of dilute nitric acid. Finally wash the precipitate with absolute alcohol and dry at 110°C to a constant mass. Preserve the filtrate for further tests ( *see* **4.2** ).

**A-3.3 Calculation**

$$\text{Palladium ( as Pd ), percent by mass} = \frac{31.61 M_1}{M}$$

where

 $M_1$  — mass in g of the dry precipitate, and $M$  = mass in g of the material taken for the test ( **A-2.1** ).**A-4. TEST FOR SILVER**

**A-4.0 General** — Silver gives a red-violet precipitate with *p*-dimethyl-amino-benzylidene-rhodanine ( *p*-dimethyl-amino-benzol-rhodanine ) in an acidic medium. Interference by metal ions like mercury, copper, gold, palladium and lead may be prevented by treating the precipitate with 5 percent potassium cyanide solution and filtering.

**A-4.1 Reagents****A-4.1.1 Concentrated Nitric Acid** — *See* IS : 264-1976\*.**A-4.1.2 Concentrated Sulphuric Acid** — *See* IS : 266-1977†.\*Specification for nitric acid ( *second revision* ).†Specification for sulphuric acid ( *second revision* ).

**A-4.1.3 Potassium Cyanide Solution** — 5 percent ( *m/v* ).

**A-4.1.4 *p*-Dimethyl-amino-benzylidene-rhodanine solution** — Dissolve 30 mg of the reagent in acetone and make up to 100 ml.

**A-4.1.5 Dilute Nitric Acid** — 10 percent ( *v/v* ).

**A-4.2 Procedure** — Take the filtrate reserved in **A-3.2** and add 5 ml of concentrated nitric acid and 5 ml of concentrated sulphuric acid. Evaporate on a sand bath to heavy fumes of sulphur trioxide. If the residue after fuming is discoloured by carbon, add cautiously 5 ml of concentrated nitric acid and fume again to dryness. Cool the residue and dilute to 25 ml.

**A-4.2.1** Take 5 ml of the solution. Add 2 ml of potassium cyanide solution and 1 ml of *p*-dimethyl-amino-benzylidene-rhodanine solution. Then stir in 2 ml of dilute nitric acid.

**A-4.2.2** The material shall be taken as having satisfied the requirement of the test if no red-violet precipitate is formed.

## **A-5. TEST FOR LEAD**

**A-5.0 General** — A neutral solution of lead develops a brick-red colour when shaken with dithizone solution in carbon tetrachloride.

### **A-5.1 Reagents**

**A-5.1.1 Dithizone Solution** — Dissolve 1 to 2 mg of the reagents in 100 ml of carbon tetrachloride. Prepare freshly before use.

**A-5.2 Procedure** — Take a drop of the solution prepared in **A-4.2**, in a small test tube, add a drop of dithizone solution and shake vigorously.

**A-5.2.1** The material shall be taken as having satisfied the requirement of the test if no brick-red colour develops.

## **A-6. TEST FOR COPPER**

**A-6.0 General** —  $\alpha$ -Benzoin oxime ( cupron ) gives a green precipitate with copper in dilute ammoniacal solution. Metal ions which are precipitated by ammonium hydroxide are kept in solution by the addition of sodium potassium tartrate to make the reagent specific for copper.

### **A-6.1 Reagents**

**A-6.1.1 Dilute Ammonium Hydroxide** — 1 : 4 ( *v/v* ).

**A-6.1.2 Sodium Potassium Tartrate Solution** — 10 percent (*m/v*) in water.

**A-6.1.3  $\alpha$ -Benzoinoxime Solution** — 5 percent (*m/v*) in alcohol.

**A-6.2 Procedure** — Take a drop of weekly acid solution on a filter paper, add a drop of sodium potassium tartrate followed by a drop of  $\alpha$ -benzoinoxime solution and hold over ammonia.

**A-6.2.1** The material shall be taken as having satisfied the requirement of the test if no green coloration is produced.

## A-7. TEST FOR IRON

**A-7.0 General** — Aqueous solution of 7-iodo-8-hydroxyquinoline-5-sulphonic acid (iodoxy-quinsulfonic acid or loretin) gives a stable green colour with ferric ions; the intensity of colour varies with the concentration of ferric ions.

### A-7.1 Reagents

**A-7.1.1 Buffer solution** — Prepare buffer of pH 2.5 by mixing 25 ml of 0.2 *M* potassium hydrogen phthalate (40.83 g per litre solution) with 36.3 ml of 0.1 *N* hydrochloric acid (10 ml of concentrated hydrochloric acid diluted with one litre water) and making them up to 100 ml in a flask.

**A-7.1.2 7-Iodo-8-Hydroxyquinoline-5-Sulphonic Acid Solution** — 0.1 percent (*m/v*) in water.

**A-7.2 Procedure** — Take a drop of the solution prepared in A-4.2 on a spot plate, add a drop of buffer solution followed by a drop of 7-iodo-8-hydroxyquinoline-5-sulphonic acid solution and mix.

**A-7.2.1** The material shall be taken as having satisfied the requirement of the test if no green coloration is produced.

## APPENDIX B

( Clause 4.1 )

### SAMPLING OF PALLADIUM SALTS FOR ELECTROPLATING

#### B-1. GENERAL REQUIREMENTS OF SAMPLING

**B-1.0** In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

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**B-1.1** Samples shall be taken in a protected place not exposed to damp air, dust or soot.

**B-1.2** The sampling instrument shall be clean and dry.

**B-1.3** Precaution shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

**B-1.4** To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

**B-1.5** The samples shall be placed in suitable, clean, dry and air-tight containers on which the material has no action.

**B-1.6** The sample containers shall be of such a size that they are almost completely filled by the sample.

**B-1.7** Each sample container shall be sealed air-tight after filling, and marked with full details of sampling and the date of sampling and details given in 3.2.

**B-2. SCALE OF SAMPLING**

**B-2.1 Lot** — All the containers in a single consignment of the material of the same type drawn from a single batch of manufacture shall constitute the lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

**B-2.2** For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot separately.

**B-2.3** The number of containers to be selected at random from a lot shall depend upon the lot size and shall be in accordance with col 1 and 2 of Table 2.

**B-2.3.1** In order to ensure the randomness of selection, procedures given in IS : 4905-1968\* shall be followed.

**B-3. INDIVIDUAL SAMPLES AND COMPOSITE SAMPLES**

**B-3.1** From each of the containers selected as in **B-2.3**, a representative portion of the material shall be drawn using a suitable sampling

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\* Methods for random sampling.

implement. The total quantity so drawn from each of the containers shall be approximately equal to thrice the quantity required for testing purpose.

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**TABLE 2 SCALE OF SAMPLING**

( Clause B-2.3 )

| LOT SIZE      | NUMBER OF CONTAINERS<br>TO BE SELECTED |
|---------------|----------------------------------------|
| ( <i>N</i> )  | ( <i>n</i> )                           |
| ( 1 )         | ( 2 )                                  |
| Up to 25      | 2                                      |
| 26 „ 50       | 3                                      |
| 51 „ 100      | 4                                      |
| 101 and above | 5                                      |

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**B-3.2** From the samples ( **B-3.1** ) representing different containers a small but approximately equal quantity of the material shall be taken and mixed thoroughly to form a composite sample sufficient to carry out testing of the characteristics as specified. The composite sample so obtained shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

**B-3.3** The remaining portion of the material in sample ( **B-3.1** ) from different containers shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the containers selected shall be for the purchaser, another for the supplier and the third for the referee.

**B-3.4** All the individual and composite samples shall be transferred to separate containers. These containers shall then be sealed air-tight and labelled with full identification particulars given in **B-1.7**.

**B-3.5** A referee sample consisting of the composite sample and a set of individual samples shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. This shall be used in case of any dispute between the two.

**B-4. NUMBER OF TESTS AND CRITERIA FOR CONFORMITY**

**B-4.1** Test for the determination of palladium ( as pd ) shall be carried out on any two of the individual samples.

**B-4.2** Tests for the remaining characteristics shall be done on the composite sample.

**B-4.3** The lot shall be deemed to conform to this specification if the test results on individual and composite samples satisfy the corresponding requirements given in Table 1.